NAVORD REPORT 6241

DEVELOPMENT OF AN OZONE RESISTANT SYNTHETIC RUBBER COMPOUND FOR LOW TEMPERATURE SERVICE (U)

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4 DECEMBER 1958

U. S. NAVAL ORDNANCE LABORATORY
WHITE OAK, MARYLAND

DEVELOPMENT OF AN OZONE RESISTANT SYNTHETIC RUBBER COMPOUND FOR LOW TEMPERATURE SERVICE

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ABSTRACT: A program to develop rubber diaphragms for the Hydrostatic Switch XH-46C was carried out. Various formulations based on a styrene-butadiene polymer were compounded and evaluated for low temperature flexibility, aging, and ozone resistance under outdoor and accelerated laboratory environmental conditioning. Formulation WA-27 was initially selected as the diaphragm material for the first production switches. When it proved to have inadequate ozone resistance properties, a special antiozonant chemical coating was found which would give adequate protection for the diaphragms already in service. Later, Formulation WA-36, an ozone resistant material, was also found to meet the requirements set forth for a diaphragm material. Formulation WA-36 is recommended for use in the manufacture of diaphragms for the Hydrostatic Switch XH-46C. This material is covered by specification NAVORD OS 7887.

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This report covers the development and evaluation of an ozone resistant synthetic rubber compound for low temperature service. The rubber is fabricated in the form of a diaphragm and serves the dual function of providing a water tight seal as well as a pressure transfer medium for actuation of the piston of the Hydrostatic Switch XH-46C. The purpose of this report is to assemble the information obtained and to make it available to other activities. This work was performed under Task 711-564/42004/01040.

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By direction

ACKNOWLEDGEMENTS

The authors wish to express their thanks and to acknowledge the cooperation of Rock Island Arsenal. Messers Shaw, Ossefort and Bergstrom conducted the ozone tests and made helpful suggestions on formulating styrene-butadiene polymers for ozone resistance.

REFERENCES

a. NAVORD OS 7887, Descriptions and Requirements, Synthetic Rubber Compound, Butadiene-Styrene Type, Ozone Resistant, for Low Temperature Service

b. Rock Island Arsenal Technical Report No. 56-3158, Protection of Rubber Vulcanizates from Ozone Cracking by External Antiozonant Application, by E. W. Bergstrom, 1 November 1956 AD-117 549

c. Federal Test Method Standard No. 601, Rubber: Sampling and Testing

d. ASTM D518-44, Method B (Bent Loops)

e. ASTM D1149-55T, Accelerated Ozone Cracking of Vulcanized Rubber

f. MIL-STD-304, JAN Temperature and Humidity Cycle

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INTRODUCTION

1. A program to develop rubber diaphragms for the Hydrostatic Switch XH-46C was carried out. Two diaphragms are utilized in this application, one outside the switch where it is exposed to all environmental conditions and the other inside where it is protected. It was the goal of this development to provide a diaphragm material with the following characteristics:

a. Compatibility with silicone grease under all environmental conditions at temperatures ranging from -54°C to 71°C.

b. Excellent flexibility at -54°C in order to maintain switch actuation pressures within required limits over the temperature range of -54°C to 71°C.

c. Adequate tensile strength, elongation and tear resistance for the application.

d. Good aging properties including ozone resistance.

e. Capability of withstanding 1,000 actuations without rupture or leakage in the hydrostatic switch.

f. Maintenance of the above properties for a storage period of 18 months minimum.

2. The dual diaphragm system for the Hydrostatic Switch XH-46C is illustrated by Figure 1. In this switch the outer diaphragm is used primarily to prevent icing around the piston area and as a safety measure to prevent rupture of the inner diaphragm. Only the outer diaphragm is exposed directly to the atmosphere. Convolutions were molded into this outer diaphragm for the first production switches. The inner diaphragms were molded without convolutions and required folding as shown in Figure 1. Both inner and outer diaphragms, subsequently, have been molded with convolutions so that in their normal position, in storage, they will not be under significant stress.

3. This report describes the development of rubber formulations based on a styrene-butadiene polymer selected for its low temperature flexibility properties. Data on physical properties and ozone resistance under outdoor and accelerated laboratory environmental conditioning are included. The application of antiozonant coatings to molded rubber surfaces is discussed. A specification, NAVORD OS 7887 (reference (a)), covering the physical properties and ozone resistance of rubber Formulation WA-36 has been written.

DEVELOPMENT WORK

4. The selection of a base material for rubber diaphragms was governed primarily by requirements for compatibility with silicone grease, low temperature flexibility, and tear resistance. Styrene-butadiene polymers were selected as the base material in trial formulations because of their low temperature flexibility properties. A selection of seven of the trial formulations pertinent to the following discussions are given in Table I.

5. Formulation WA-4 had been used successfully in contact with silicone fluid for a previous application. Therefore, WA-4 was selected for the initial trial formulation but its tear resistance proved inadequate for diaphragm requirements, and its tensile strength and elongation were lower than desired. Formulation WA-7 incorporated Philblack O in order to obtain higher tensile strength properties, and Hi-Sil C was added to improve the tear resistance. The base material was then changed to SBR 1023 in Formulation WA-9 to further increase the tensile strength.

6. In order to improve the low temperature flexibility, the amount of plasticizer (TP-90B) was increased in Formulation WA-10. Further study was made on WA-10 to determine aging properties and long term effects of contact with silicone grease. This material appeared to be adequate for the requirements set forth, and the formulation was released for production of rubber diaphragms. The contractor for molding diaphragms, however, was unable to obtain Hi-Sil C on the commercial market. Further study indicated Hi-Sil 233 could be used as an adequate substitute for Hi-Sil C, and Formulation WA-27 was, therefore, used as the material in the first production switches.

7. The evaluation of Formulation WA-27 consisted of tests on molded diaphragms as well as on standard test panels. Two diaphragm shapes were used for tests. These are designated as the inner and outer diaphragms of the Hydrostatic Switch XH-46C and are shown in Figure 1. Both the inner and outer diaphragms were tested for ozone resistance as well as for rupture resistance. Rupture resistance was determined by inflating the diaphragms with air after various environmental exposures. Two groups of diaphragms were used for these tests. During exposure, one group of diaphragms were mounted in the hydrostat housings using silicone grease to fill the cavity between the inner and outer diaphragms as shown in Figure 1. These diaphragms were designed as "Mounted" in Table II. Another group of diaphragms were neither mounted in the switch housing nor coated with silicone grease. These diaphragms were subjected to various exposures in the "as molded" condition and are designated "Unmounted" in Table II. Diaphragms exposed to outdoor exposure were not coated with silicone grease.

8. Formulation WA-27 was found to be susceptable to ozone attack, and, therefore, was considered inadequate for the diaphragm application.

Other properties of this material were satisfactory. According to reference (b), it is possible to protect SBR vulcanizates against ozone attack by externally coating the molded specimens twice with a 50/50 parts by volume UOP-88/acetone solution. It was determined that those diaphragms of Formulation WA-27 now in service could be adequately protected in this manner.

9. Additional work was undertaken to obtain a rubber formulation with improved ozone resistance and variations in the amount of antiozonant and wax carrier resulted in the development of Formulation WA-36 as an ozone resistant material. As indicated by Table III, the properties of Formulation WA-36 are as good or better than WA-27. Therefore, all experimental and production diaphragms are currently being molded of Formulation WA-36.

TEST METHODS

10. Standard test methods, references (c), (d) and (e), were used to evaluate panel stocks for their mechanical properties. Details are given in reference (a).

11. Two tests were made to determine the low temperature flexibility. The first of these, the brittleness test, consists of maintaining specimens at -54°C for four hours and then allowing a 65 gram steel ball to fall 37 cm onto the rubber test strip. This strip is held by one end, fastened between two wooded blocks, with its other end placed perpendicular to and in the path of the falling ball. A brittle material will crack on impact, a nonflexible material will hold the ball in the guide tube, and a flexible material will bend to release the ball from the tube. The second test was the Gehman low temperature flexibility test as described in Method 5611 of reference (c).

12. Ozone resistance was another important property which was evaluated on the most promising formulations. Specimens for ozone resistance testing were mounted in accordance with reference (d). Some specimens were exposed to an accelerated test in an ozone cabinet, as described in reference (e), but with an ozone concentration of 50 ± 5 parts per hundred million and a temperature of 37.8 ± 1.1°C. Specimens were examined under twenty power magnification at intervals during the seven days test period. All accelerated ozone resistance testing was performed by Rock Island Arsenal, Rock Island, Illinois. Other ozone resistance test samples were mounted for outdoor exposure on Lucite racks as shown in Figure 2. These racks were mounted at a 45° angle facing South at the Canal Zone Corrosion Laboratory, Miraflores, Panama Canal Zone, and at the Naval Ordnance Laboratory, White Oak, Silver Spring, Maryland. The rack exposed at the Naval Ordnance Laboratory Test Facility, Fort Lauderdale, Florida, was mounted at a 45° angle facing parallel to the edge of the ocean (due East). Specimens exposed on the roof at the Naval Ordnance Laboratory were examined at random intervals under eight power magnification.

13. The effect of various laboratory conditioning tests on rubber materials is usually determined by the change in physical properties of

standard specimens. This type of testing, however, was not feasible for molded diaphragms. Therefore, a test was devised which would measure the air pressure required to rupture a diaphragm. Figure 3 illustrates this test set-up. A modified diaphragm housing, similar to the hydrostatic switch, was used with the diaphragms mounted as shown in Figure 3. To test an inner diaphram, an initial air pressure of twelve psi was applied, and then increased by two psi steps at fifteen second intervals until a pressure of 28 psi was reached. The pressure was then increased one psi steps at fifteen second intervals until rupture of the diaphragm occurred. When testing the outer diaphragm, an O-ring was used to replace the inner diaphragm. An initial air pressure of six psi was used. Increased pressures were applied in two psi steps each fifteen seconds up to ten psi. Subsequent increases in pressure were at one psi steps at fifteen second intervals until rupture of the diaphragm occurred.

TEST RESULTS

14. Test results discussed herein are limited to the evaluation of Formulation WA-27 and Formulation WA-36. A summary of the physical properties of these two materials is given in Table III.

15. Low temperature flexibility of the rubber diaphragm is extremely important if actuation pressures are to be maintained within required limits over the temperature range of -54° to 71°C. The Gehman low temperature flexibility test results are illustrated by Figure 4. A twist of 73 degrees at -54°C for Formulation WA-27 indicates that this rubber has good compliance at low temperature. The comparable curve for Formulation WA-36 shows WA-36 to be more flexible than WA-27 in the temperature range. Repeated tests on various batches indicated WA-36 to be consistently more flexible than various batches of WA-27 at -54°C.

16. Ozone resistance testing was conducted at 37.8°C and at 50 parts per hundred million ozone concentration. Slight cracking was noted within two hours when specimens of Formulation WA-27 were examined under twenty power magnification. Small cracks over the top surfaces of the bent loops were visible to the naked eye when specimens were removed after seven days exposure (see Figure 5). Similar specimens for Formulation WA-36 (Figure 5) passed seven days of ozone exposure without visible cracking as examined under twenty power magnification.

17. Bent loop specimens of Formulations WA-27 and WA-36 were subjected to outdoor exposure. Cracks were visible to the naked eye on WA-27 bent loops after four weeks exposure in April on the roof of the Naval Ordnance Laboratory. As illustrated by Figure 6 all WA-27 specimens had cracked during outdoor exposure in various locations.

Bent loops of Formulation WA-36 remained crack free (examined under seven power magnification) after six months of outdoor exposure on the roof of the Naval Ordnance Laboratory (see Figure 7).

18. Molded diaphragms of Formulation WA-27 were subjected to outdoor exposure and ozone resistance testing. The condition of unmounted diaphragms after outdoor exposure is shown in Figure 8. The stressed position of the inner diaphragm accelerated cracking at the stressed areas while the outer diaphragms were unstressed and only slight cracking appeared in the top center of the diaphragm. As illustrated by Figure 9, the ozone cabinet tests were more severe than outdoor exposure tests. Numerous cracks were visible within 24 hours as shown by Diaphragm No. 13, and within seven days the stressed wall had cracked through (No. 14). The stressed outer diaphragm was in several pieces after seven days exposure. Ozone tests on outer diaphragms fabricated of WA-27 mounted in the hydrostatic switch housing and filled with silicone grease showed only a slight evidence of ozone cracking after seven days. The inner diaphragms in this same test were unaffected. Ozone atmosphere was kept from contact with the exposed side of the inner diaphragm by a plate sealed to the bottom of the housing (see Figure 1).

19. Stressed diaphragms of Formulation WA-36 remained crack free (examined under seven power magnification) after six months of outdoor exposure on the roof of the Naval Ordnance Laboratory. The improved ozone resistance of Formulation WA-36 during outdoor exposure tests is illustrated by Figure 7 as compared to Figures 6 and 8 which show results of WA-27 exposures.

20. Bent loop specimens and outer diaphragms (molded of WA-27) which had been coated with a UOP-88/acetone solution prior to exposure remained crack free for seven days ozone resistance testing at 50 ± 5 parts per hundred million ozone concentration and 37.8°C (see Figures 5 and 9). Since weather conditions affect outdoor exposure tests, samples were simultaneously exposed coated and uncoated at the Naval Ordnance Laboratory for direct comparison of cracking effects. After six months outdoor exposure, uncoated specimens were cracked as illustrated by Figure 10. Coated specimens had not cracked, although the UOP-88 coating had checked on the surface (see Figure 10).

21. The results of the rupture test of diaphragms molded of Formulation WA-27 are given in Table II. The hydrostatic switch is required to pass a JAN Cycle test, (reference (f)), to demonstrate its resistance to deterioration under service environmental conditions. This test consists of 28 days, or two complete cycles. Each cycle exposes the samples to temperature-humidity extremes as illustrated by Figure 11. Rupture test results indicated less change as a result of JAN Cycle than that obtained by oven aging or outdoor exposure. Six months additional outdoor exposure in Panama did not significantly change the rupture test values from those obtained after the first

six months exposure. Rupture testing of diaphragms conditioned with or without contact with silicone grease did not give sufficient evidence of adverse aging of diaphragms molded of Formulation WA-27. Test results did not warrant additional rupture testing using diaphragms molded of Formulation WA-36.

CONCLUSIONS

22. Two synthetic rubbers have been formulated for use in the Hydrostatic Switch XH-46C application. Formulation WA-27 was used as the diaphragm material for the first production switches but was later found to be susceptable to deterioration by ozone during adverse storage conditions. It was further found that this shortcoming could be overcome by twice coating the material with a 50/50 parts by volume UOP-88/acetone solution. This technique is recommended for protection of the WA-27 diaphragms currently in service.

23. Synthetic rubber Formulation WA-36 is an improvement of WA-27 and adequately meets the requirements for the diaphragm application. It is, therefore, recommended for use in the manufacture of all subsequent diaphragms for the Hydrostatic Switch XH-46C. A specification, NAVORD OS 7887 (reference (a)), covering the physical properties of rubber Formulation WA-36 has been prepared based upon the data given in Table III.

TABLE I

ARCTIC TYPE SBR POLYMER FORMULATIONS

Formulation Designation	WA-4	WA-7	WA-9	WA-10	WA-27	WA-36
Compounding Ingredients						
SBR 1015	70	70	-	-	-	-
SBR 1505	30	30	-	-	-	-
SBR 1023	-	-	100	100	100	100
Kosmos 20	60	-	-	-	-	-
Philblack O	-	40	40	40	40	40
Hi Sil C	-	20	20	20	-	-
Hi Sil 233	-	-	-	-	20	20
TP-90B	5	5	5	15	15	15
Zinc Oxide (Kadox 25)	3	3	3	3	3	3
Stearic Acid N. F.	1.5	1.5	1.5	1.5	1.5	1.5
Sunproof Jr.	2	-	2	2	2	-
Heliozone	-	2	-	-	-	1
UOP-88	2	2	2	2	2	5
Neozone D	0.5	0.5	0.5	0.5	0.5	0.5
Sulfur (Blackbird)	0.7	0.7	0.7	0.7	0.7	0.7
Monex	2	2	2	2	2	2
D.P.G.	0.4	-	0.4	0.4	0.4	0.4
Physical Properties						
Tensile Strength, psi	1180	1330	1848	1574	1612	1544
Elongation, %	380	444	423	430	454	475
Hardness, Shore						
A*, R. T.	55	57	60	58	55	55
-54°C	60	75	75	70	70	72
Tear Resistance, lb/in	-	-	-	-	150	165
Ozone Resistance, 50 pphm, 38°C, 7 days	-	-	-	-	2 hrs.*	O.K.

* Time to first crack noted under 20 X magnifications.

TABLE II

RUPTURE TEST RESULTS OF HYDROSTATIC SWITCH DIAPHRAGMS
Molded of Formulation WA-27

Type of Conditioning	Inner Diaphragm		Outer Diaphragm	
	Conditioned Unmounted (psi)	Conditioned Mounted$_a$ (psi)	Conditioned Unmounted (psi)	Conditioned Mounted$_a$ (psi)
Unconditioned	30, 30	-	14, 14	-
Ozone, 50 pphm, 38°C, 7 days	-	33, 33, 33	-	13, 15, 13
Oxygen Bomb, 70°C, 72 hours	31	34	14	16
JAN Cycle, 28 days	35, 37	32, 32	15, 16	15, 16
Aging, 100°C, 72 hours	35, 37	34, 38	17, 17	17, 18
Aging, -73.3°C 72 hours	30, 31	30, 30	14, 14	15, 14
Outdoor exposure:				
6 months:				
Silver Spring, Md.	38, 41	-	16, 17	
Ft. Lauderdale, Fla.	41, 42	-	17, 17	-
Panama Canal Zone	42, 42	-	17, 18	-
12 months:				
Panama Canal Zone	41, 43	-	18, 18	-

NOTE:
a - Diaphragms mounted in the hydrostat housing during conditioning were in contact with Silicone Grease DC-4. See Figure 1 and paragraph 7 of text.

TABLE III

COMPARISON OF PHYSICAL PROPERTIES OF FORMULATIONS WA-27 and WA-36

Properties	NAVORD OS 7887 Specification Values	Formulation WA-27 Values	Formulation WA-36 Values
TENSILE STRENGTH			
Initial	1475 psi, min	1485-1750	1528-1702
After oven aging (70 hrs at 100°C)	± 15% change, max	0 to -9.1%	-5.3 to -9.4%
ELONGATION			
Initial	400%, min	407-465	448-499
After oven aging (70 hrs at 100°C)	-20% change, max	-6.7 to -17.4%	-4.6 to + 2.9%
HARDNESS (Shore "A" Durometer)			
Initial	55 ± 5	52 - 60	52 - 56
After oven aging (70 hrs at 100°C)	60, max	58 - 59	55 - 58
After water immersion (70 hrs at 100°C)	60, max	50	51 - 53
Low temperature (after 4 hrs at -54°C)	75, max	65 - 70	72 - 75
TEAR RESISTANCE			
Initial	150 lbs/inch of thickness, min	150	158 - 187
COMPRESSION SET			
After oven aging			
(22 hrs at 70°C)	15% change, max	10.7 - 12.4	11.3 - 12.8
(70 hrs at 100°C)	20% change, max	14.2 - 16.6	12.6 - 14.6
STIFFNESS			
After 5 min at 54°C	75° twist, min	45° - 90°	86° - 96°
BRITTLENESS			
After 4 hrs at -54°C	No failures	No failures	No failures
RESISTANCE TO SILICONE GREASE			
Hardness-Shore "A" Durometer (after treatment with silicone grease)	60, max	58 - 60	58 - 60

TABLE III (Cont'd)

COMPARISON OF PHYSICAL PROPERTIES OF FORMULATIONS WA-27 AND WA-36

Properties	NAVORD OS 7887 Specification Values	Formulation WA-27 Values	Formulation WA-36 Values
RESISTANCE TO SILICONE GREASE (Cont'd)			
Thickness (after treatment with silicone grease)	-5% change, max	-2.9 to -4.3%	0 to -2.7%
Weight (after treatment with silicone grease)	-10% change, max	-5.2%	-3.9 to -8.0%
WEIGHT CHANGE			
After oven aging (70 hrs at 100°C)	-5% change, max	-1.8 to -2%	-2.7 to -3.8%
VOLUME CHANGE			
After water immersion (70 hrs at 100°C)	+5% change, max	2.1 to 3.2%	3.2 to 4.4%
CONDUCTANCE			
Water Extract	70 $ohm^{-1}cm^{-1}$ x 10^{-6}, max	65	57 - 68
SPECIFIC GRAVITY	1.15 ± 0.05	1.13 - 1.15	1.14
OZONE RESISTANCE			
After 7 days in 50 pphm ozone at 38°C	Crack free under 20X magnification	2 hours - cracked	crack free

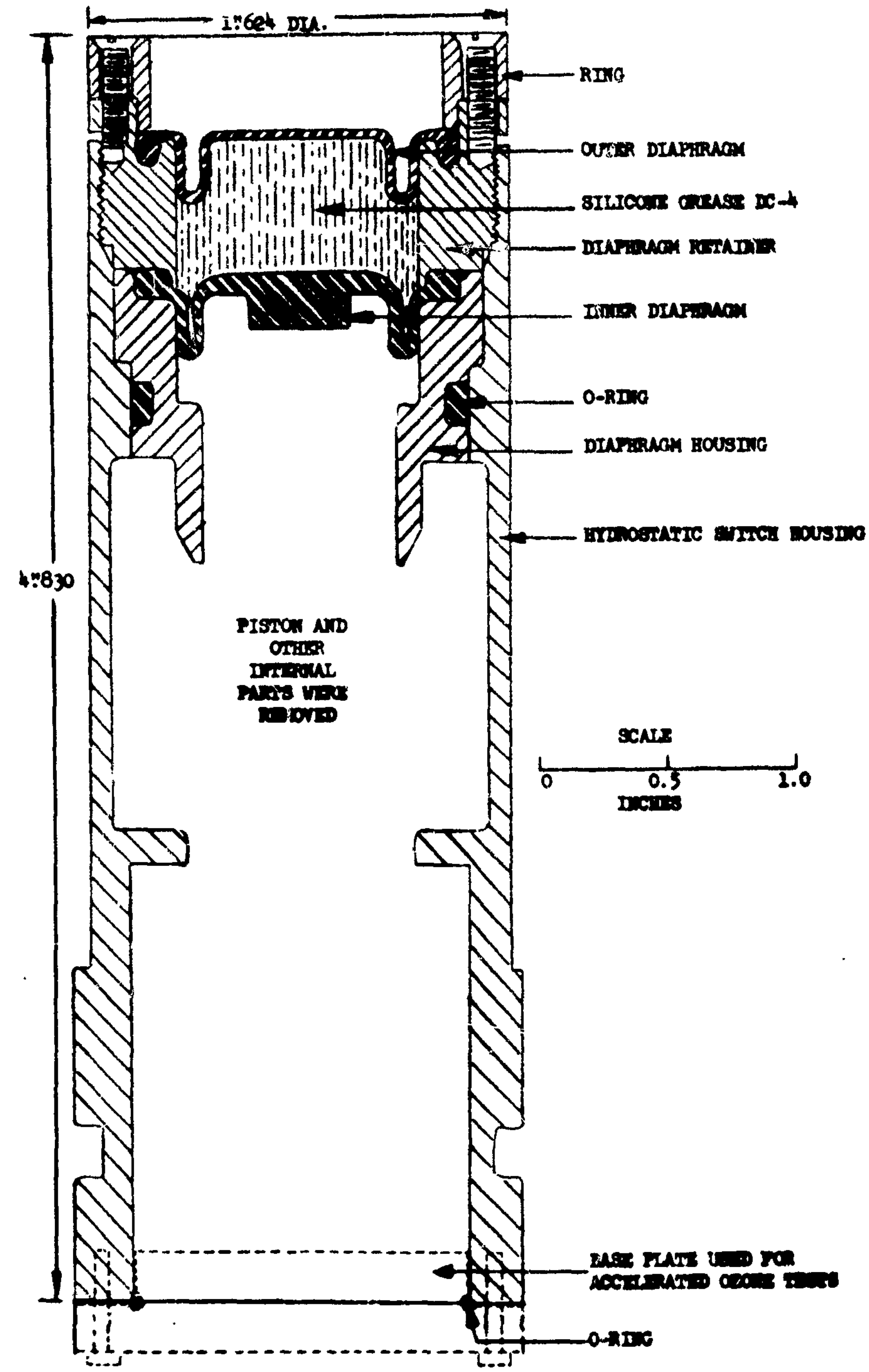


FIGURE 1. HYDROSTATIC SWITCH XH-46C, MODIFIED

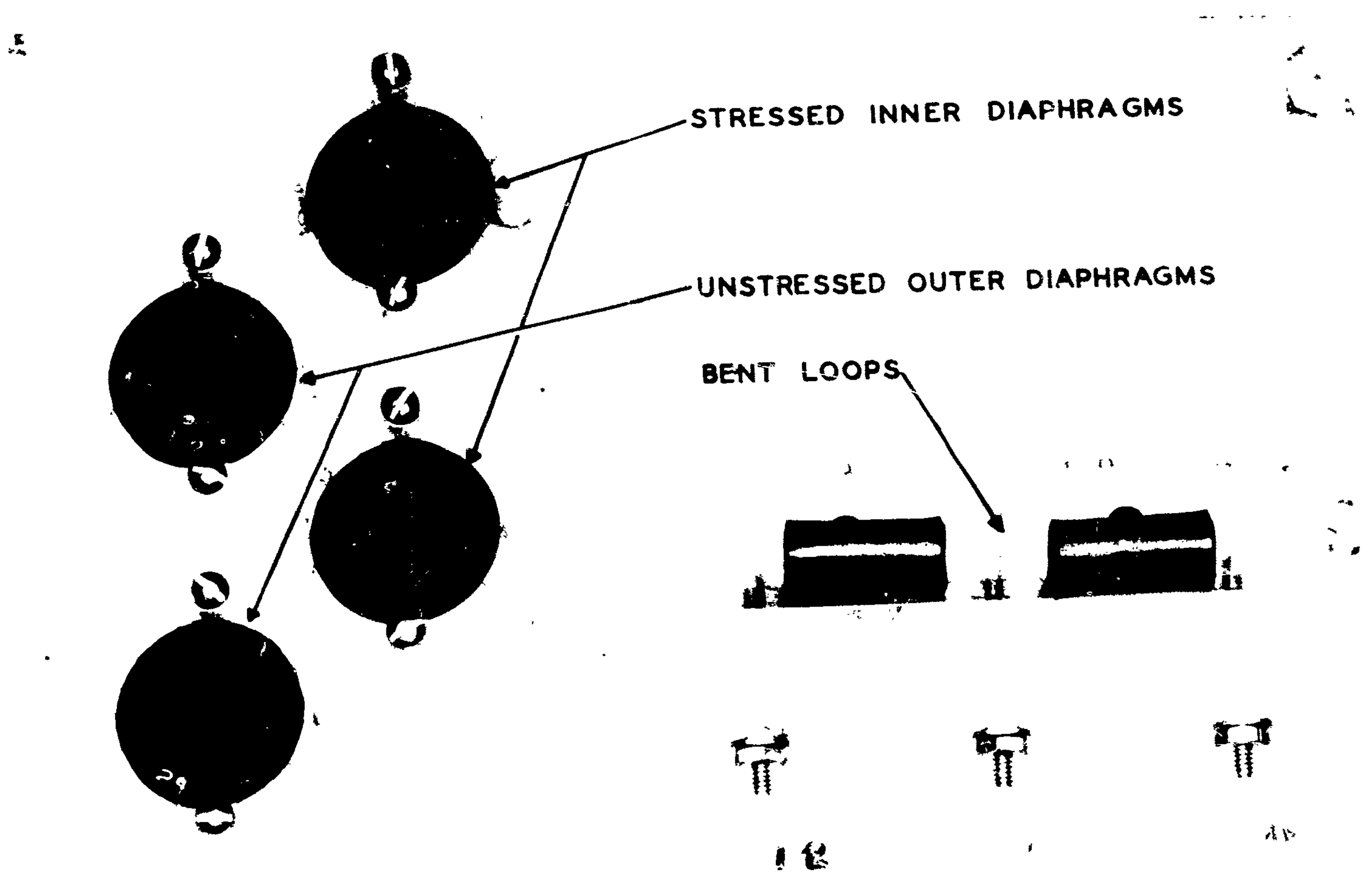


FIGURE 2. OUTDOOR EXPOSURE RACK, PANAMA CANAL ZONE, TWELVE MONTHS EXPOSURE

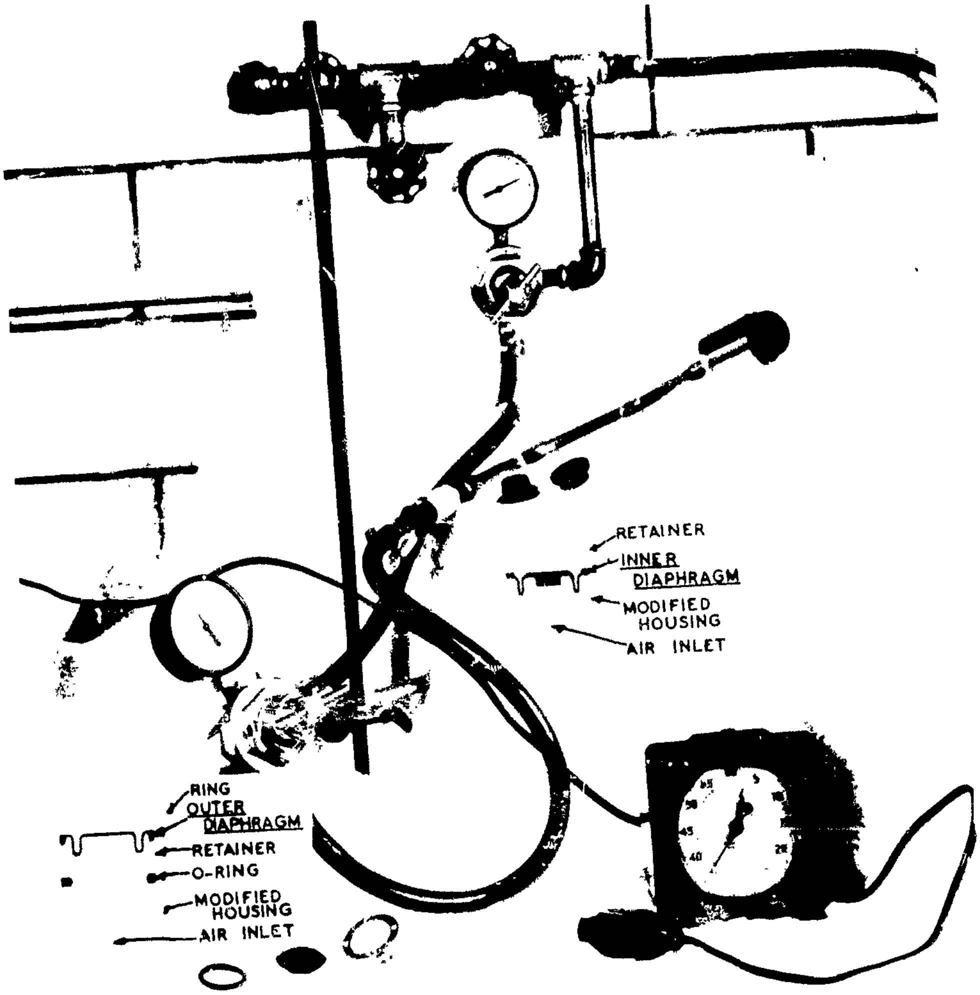


FIGURE 3. RUPTURE TEST SET UP FOR MOLDED DIAPHRAGMS

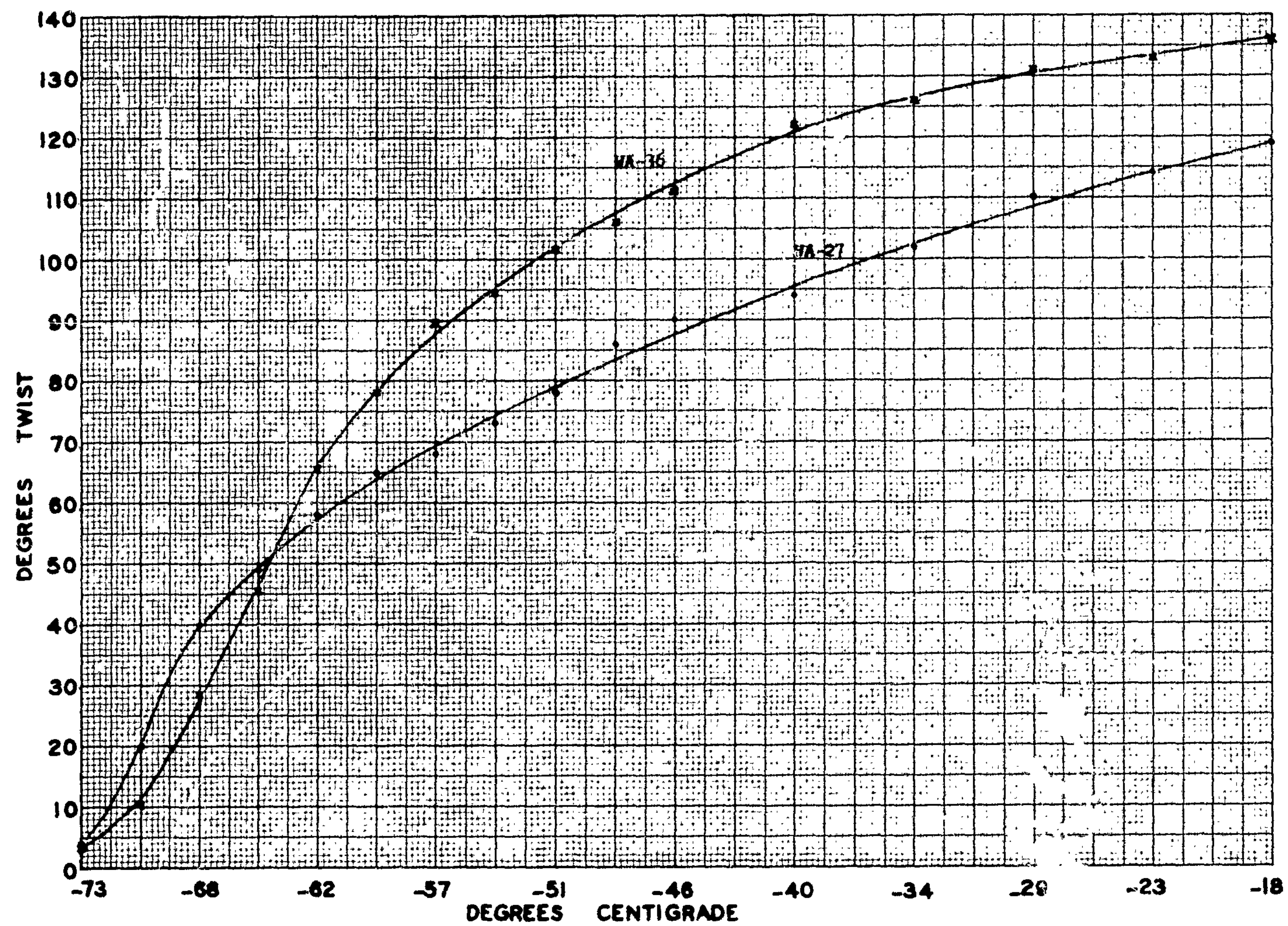
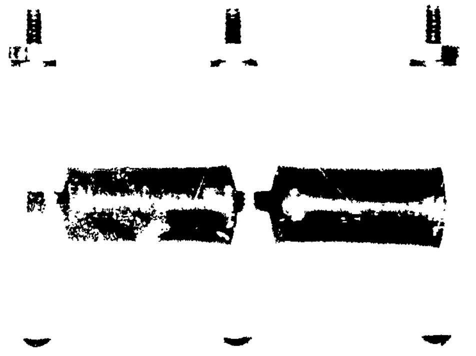
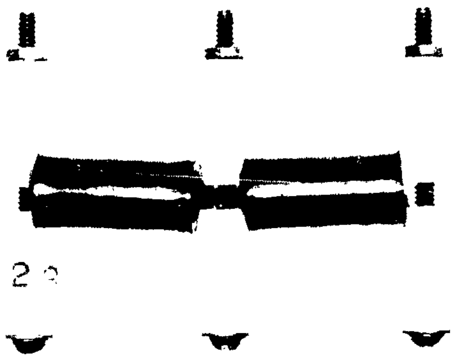


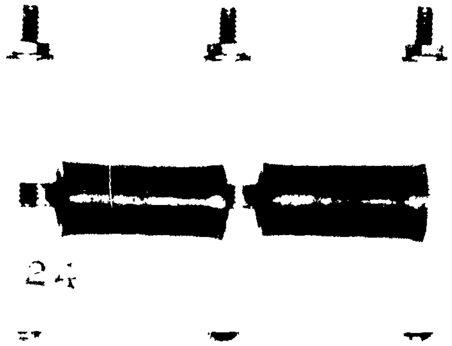
FIGURE 4. GEHMAN LOW TEMPERATURE FLEXIBILITY OF DIAPHRAGM FORMULATIONS WA-27 & WA-36



WA-27 (CHALKED TO SHOW CRACKS)
FIRST CRACK - 2 HOURS



WA-27 COATED WITH UOP-88
CRACK FREE - 7 DAYS



WA-36
CRACK FREE - 7 DAYS

FIGURE 5. OZONE RESISTANCE TEST, BENT LOOPS, 50 PPHM OZONE, 38°C, SEVEN DAYS

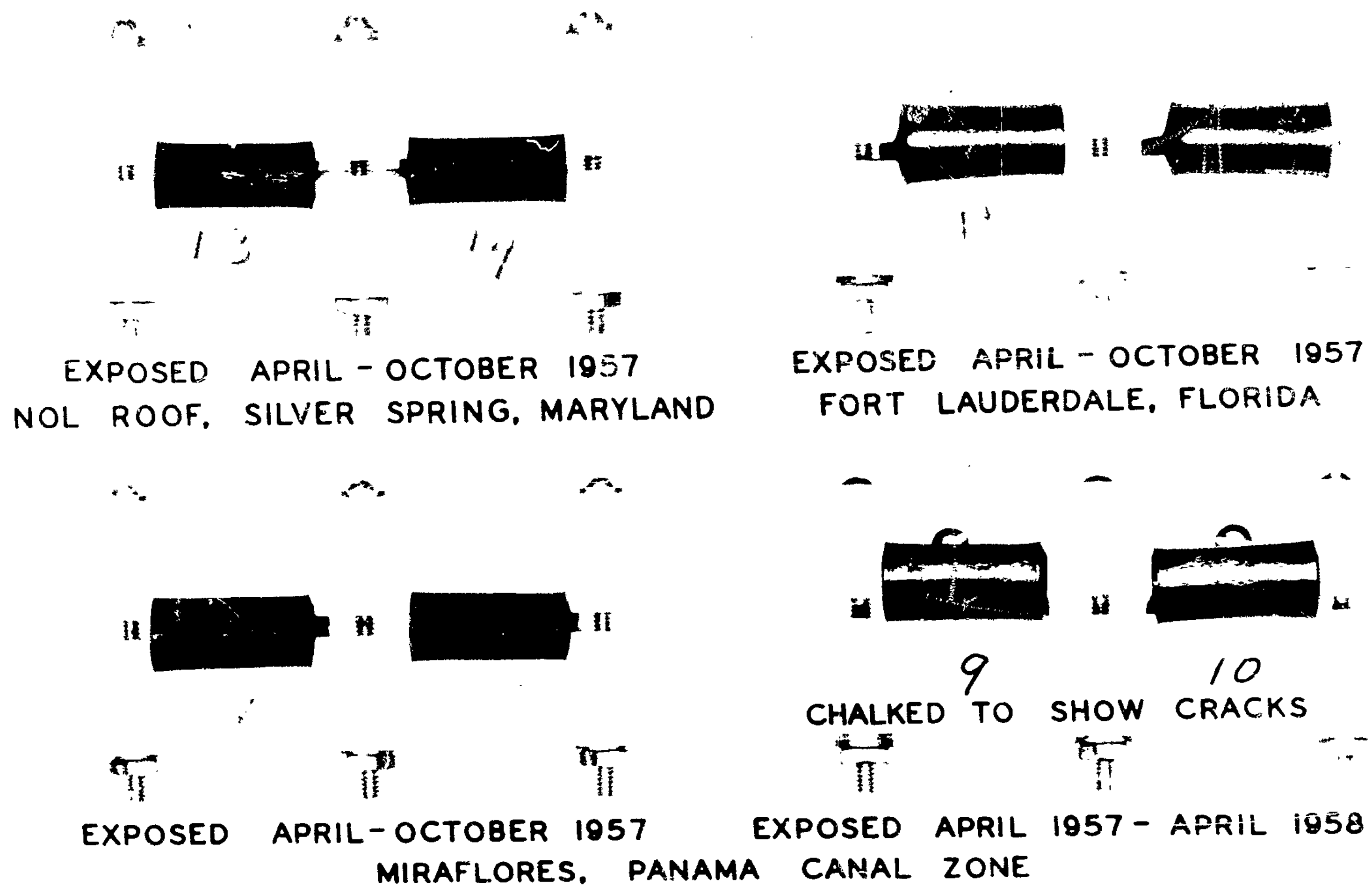
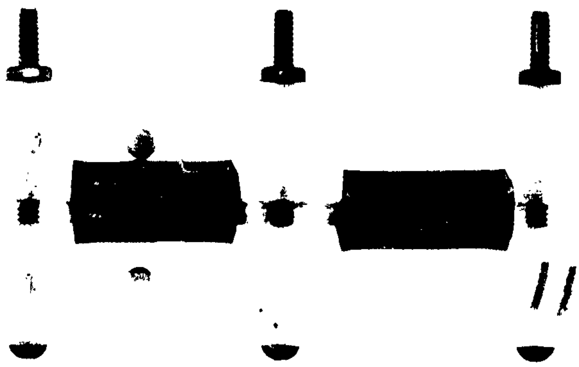
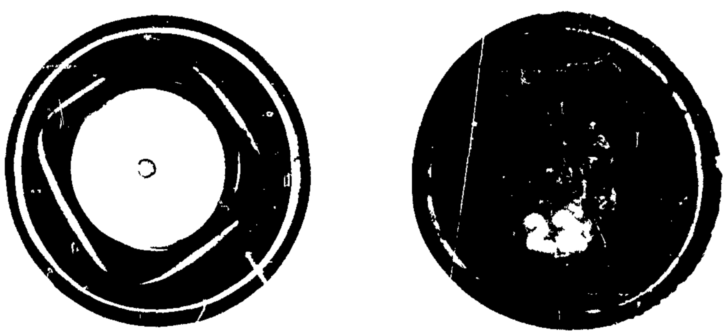


FIGURE 6. SIX AND TWELVE MONTHS OUTDOOR EXPOSURE OF WA-27, BATCH 16, BENT LOOPS



BENT LOOPS EXPOSED
4 SEPTEMBER 1957 - 4 MARCH 1958



STRESSED
INNER DIAPHRAGMS EXPOSED
11 SEPTEMBER 1957 - 11 MARCH 1958

FIGURE 7. SIX MONTHS OUTDOOR EXPOSURE ON NOL ROOF OF WA-36 BENT LOOPS AND DIAPHRAGMS

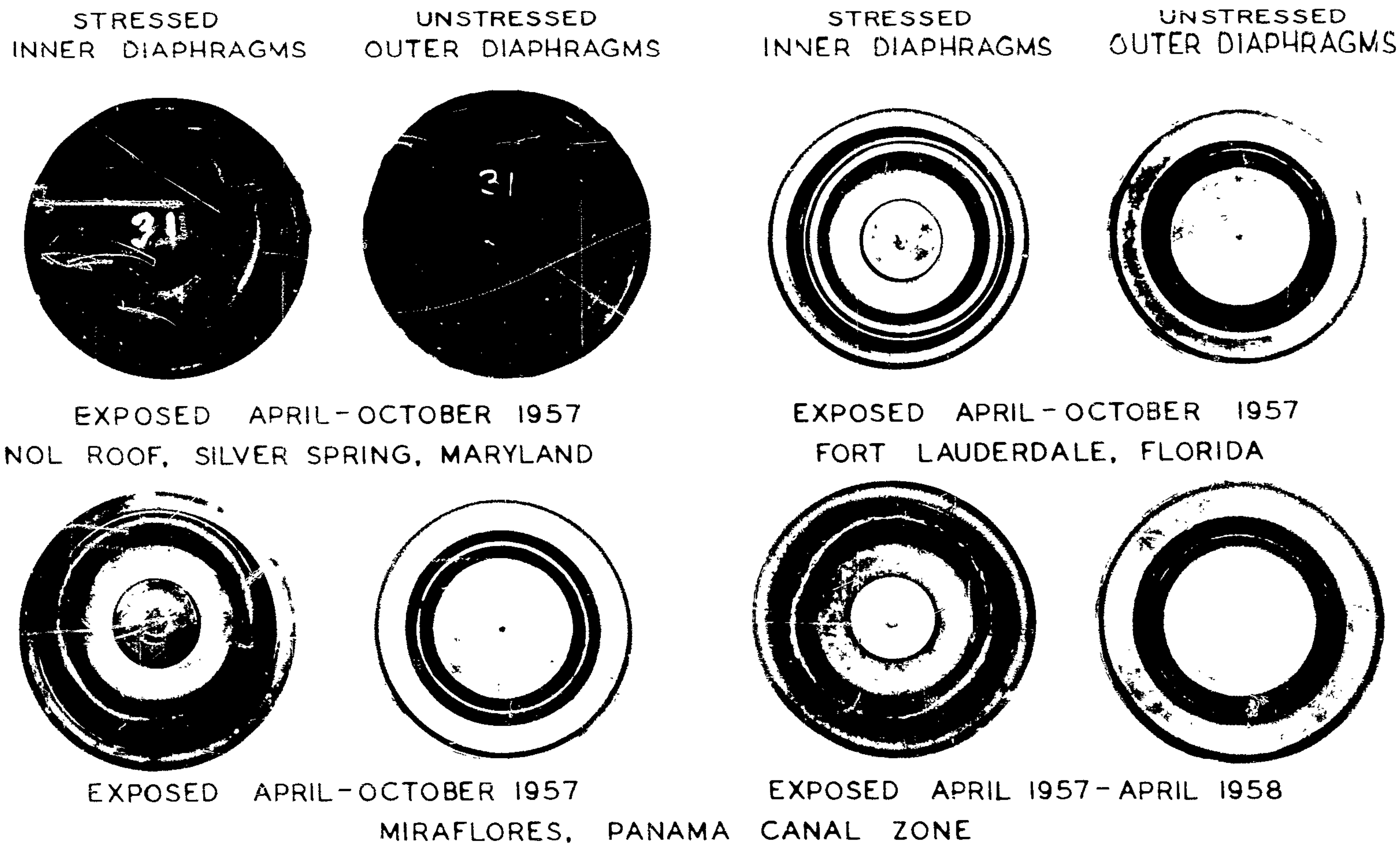
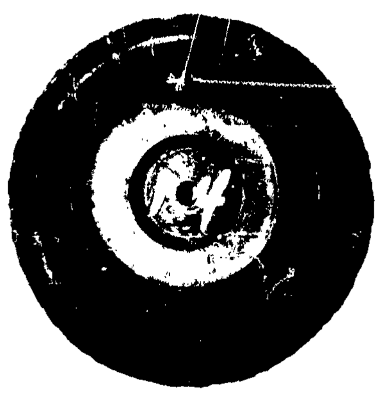


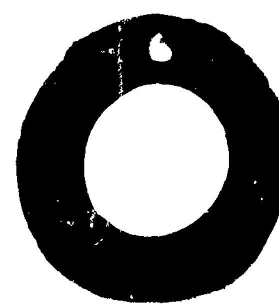
FIGURE 8. SIX AND TWELVE MONTHS OUTDOOR EXPOSURE OF WA-27, BATCH 16, MOLDED DIAPHRAGMS

INNER DIAPHRAGMS
EXPOSED AS ASSEMBLED
IN HYDROSTAT

OUTER DIAPHRAGMS
EXPOSED IN DOME SHAPE
FOR STRESS



STRESSED FOLD
CRACKED THROUGH
IN 7 DAYS



TOP OF DOME
CRACKED INTO 3 PIECES
IN 7 DAYS

CRACKED IN
24 HOURS

COATED WITH UOP-88
CRACK FREE FOR
7 DAYS

FIGURE 9. OZONE RESISTANCE TEST, MOLDED DIAPHRAGMS, WA-27, 50 PPHM OZONE, 38°C

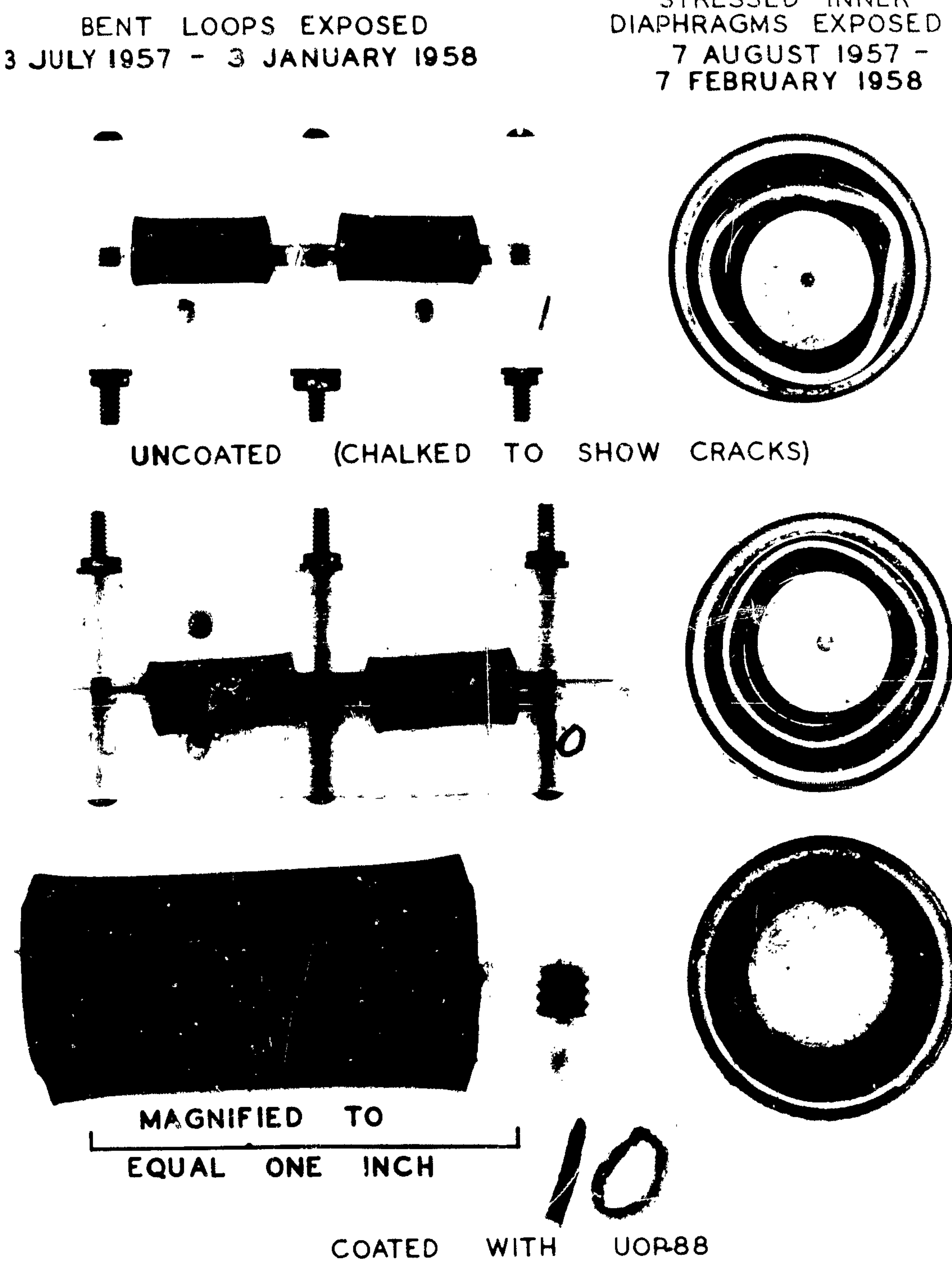
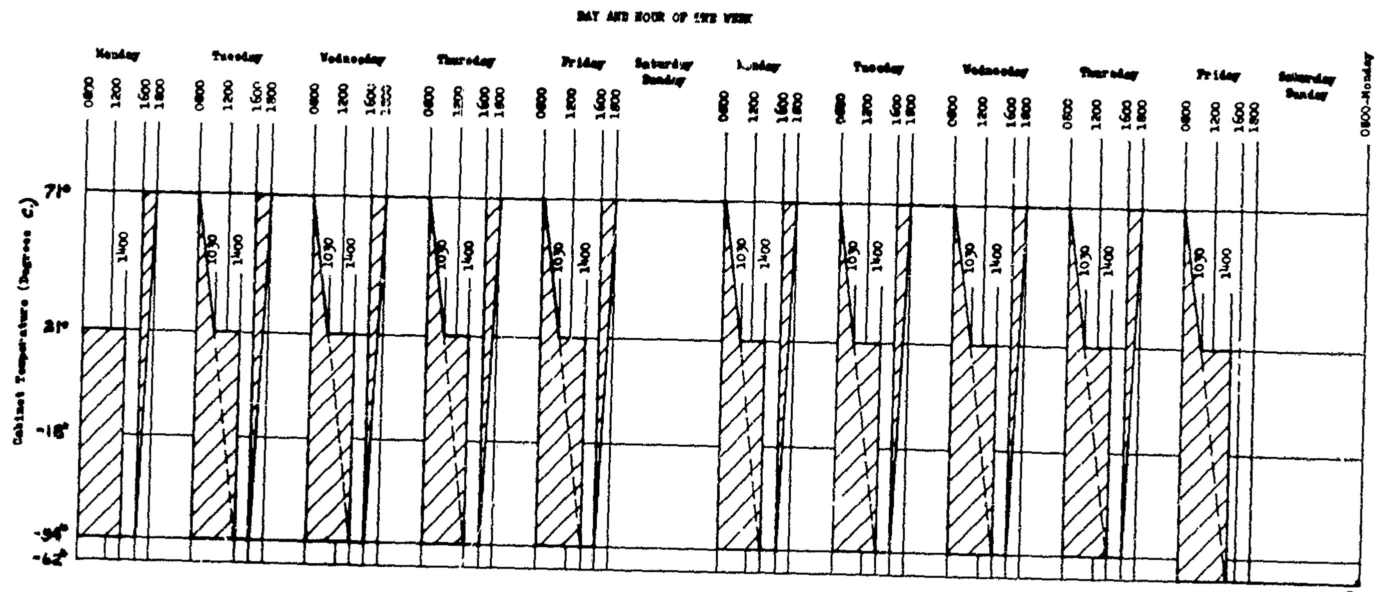


FIGURE 10. SIX MONTHS OUTDOOR EXPOSURE ON NOL ROOF OF WA-27, BATCH 21, UNCOATED AND COATED WITH UOP-88



OPERATIONAL SCHEDULE

Hour of The Day	Monday	Tuesday Wednesday Thursday	Friday (First Week-End)	Saturday (First Week-End)	Sunday (First Week-End)	Monday Tuesday Wednesday Thursday	Friday (Second Week-End)	Saturday (Second Week-End)	Sunday (Second Week-End)
0800	A	D	D	E	E	D	D	F	F
1200	A	D	D			D	D		
1400	B	B	B			B	F		
1600	C	C	C			C	F		

A - Expose fuzes to -65°F cabinet temperature before 1400.

B - Maintain cabinet temperature of -65°F until 1600.

C - Increase temperature of air surrounding fuzes to 160°F (95% R.H.) as rapidly as practicable (cabinet must be at 160°F., 95% R.H., by 1800).

D - Decrease the temperature of the air surrounding the fuzes to -65°F at an initial rate equal to or greater than 36°F per hour for the first 2 ½ hours. The rate may be changed thereafter, but shall be such that -65% F. is reached by 1400 or earlier.

E - Maintain cabinet temperature of 160°F. (95% R.H.).

F - Decrease temperature of cabinet to -80°F. This temperature to be maintained until the next operation.

FIGURE 11. JAN TEMPERATURE AND HUMIDITY CYCLE

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